and N(2) of the pyridinium ring: for molecule (I) the N(2)$\cdots$I(x, y, z) distance is 3·664 (8) Å, for molecule (II) N(2)$\cdots$I($1 - x, y - \frac{1}{2}, \frac{1}{2} - z$) is 3·590 (8) Å.

We thank Dr P. van der Sluis for preparation of single crystals and Dr A.J.M. Duisenberg for collecting the crystallographic data.

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Structure of 6-*tert*-Butyl-3-cyclohexyl-3,4-dihydro-2-(4-tolyloxy)-2*H*-1,3,2-benzoxazaphosphorine 2-Oxide

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Abstract. $C_{24}H_{32}NO_3P$, $M_r = 413{\cdot}5$, monoclinic, $P2_1/c$, $a = 9{\cdot}045\ (4)$, $b = 20{\cdot}507\ (5)$, $c = 12{\cdot}571\ (2)$ Å, $\beta = 102{\cdot}60\ (1)°$, $V = 2275{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}21$ g cm^{-3}, $\lambda(\text{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 12{\cdot}4$ cm^{-1}, $F(000) = 888$, $T = 297$ K, $R = 0{\cdot}057$ for 3178 observed reflections. The oxazaphosphorine ring adopts a conformation midway between a half-chair and a sofa. The phosphoryl O(2) atom occupies an axial position. The *tert*-butyl and phenyl groups are *trans* to each other and occupy equatorial positions on the phosphorine ring. The cyclohexyl ring adopts a chair conformation with the N atom [N(5)] equatorial. The molecular geometry is normal. The molecular packing involves only van der Waals contacts.

Introduction. The title compound was prepared and supplied by M. S. R. Naidu & Nagaraju, Department of Chemistry, S. V. University, Tirupathi, India. This type of phosphorus heterocycle has been found to possess significant antitumour activity (Chugani Pharmaceutical Co., 1966; Arnold, Bourseaux & Brock, 1961; Friedman, Papanastassiou & Levi, 1963) and for this reason its X-ray structure is of great interest to our continuing investigations.

Experimental. Transparent, colourless needle-shaped crystals were grown from ethanol. A specimen with approximate dimensions 0·30 × 0·40 × 0·55 mm was selected. Unit-cell parameters and their e.s.d.'s derived from a least-squares treatment of 25 reflections ($35 < \theta < 45°$), intensity data were collected on an Enraf–Nonius CAD-4 single-crystal X-ray diffractometer, graphite-crystal-monochromatized Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode. Intensities of reflections with $4 \leq 2\theta \leq 120°$ (h $-11 \rightarrow 10$, k $0 \rightarrow 24$, l $0 \rightarrow 15$) were measured. Two standard reflections for every 100 observations, <2% variation. Of the 3496 independent reflections collected, 3056 were judged significant [$I > 3\sigma(I)$]; intensities not corrected for absorption. Structure by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Positional and thermal parameters of non-H atoms refined by full-matrix least-squares using *SHELX*76 (Sheldrick, 1976). H atoms were located from a difference Fourier map and not refined. In the final stage of the refinement, weights were introduced resulting in $R = 0{\cdot}057$, $wR = 0{\cdot}063$, $w = 1/[\sigma^2(F) + 0{\cdot}00026F^2]$, $S = 2{\cdot}9$. $(\Delta/\sigma)_{\max} = 0{\cdot}008$. Final difference Fourier map featureless, with $\Delta\rho$ within $\pm 0{\cdot}13$ e Å^{-3}. Atomic scattering factors as provided in *SHELX*76. Computer programs: *PARST* (Nardelli, 1983) for

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
P(1)	0·3912 (1)	0·1909 (< 1)	0·9439 (1)	4·2
O(2)	0·4825 (3)	0·1561 (1)	0·8808 (2)	4·9
O(3)	0·2354 (3)	0·1572 (1)	0·9513 (2)	4·8
O(4)	0·3241 (3)	0·2568 (1)	0·8865 (2)	5·0
N(5)	0·4752 (3)	0·2084 (1)	1·0685 (2)	4·0
C(6)	0·2283 (4)	0·0975 (2)	1·0055 (2)	4·3
C(7)	0·1346 (4)	0·0951 (2)	1·0773 (3)	5·2 (1)
C(8)	0·1192 (4)	0·0372 (2)	1·1290 (3)	5·4 (1)
C(9)	0·1952 (4)	−0·0185 (2)	1·1117 (3)	4·9 (1)
C(10)	0·2880 (4)	−0·0141 (2)	1·0388 (3)	5·5 (1)
C(11)	0·3055 (4)	0·0428 (2)	0·9837 (3)	5·1 (1)
C(12)	0·3121 (4)	0·3141 (1)	0·9455 (2)	3·9
C(13)	0·2718 (4)	0·3697 (2)	0·8860 (2)	4·5
C(14)	0·2617 (4)	0·4283 (2)	0·9396 (2)	4·4
C(15)	0·2937 (4)	0·4317 (2)	1·0526 (2)	4·1
C(16)	0·3311 (4)	0·3740 (2)	1·1097 (2)	4·1
C(17)	0·3415 (3)	0·3149 (2)	1·0586 (2)	3·9
C(18)	0·3858 (4)	0·2532 (2)	1·1229 (2)	4·4
C(19)	0·6437 (4)	0·2111 (1)	1·0983 (2)	4·3
C(20)	0·7015 (5)	0·1949 (2)	1·2172 (3)	6·1 (1)
C(21)	0·8747 (5)	0·1915 (3)	1·2452 (3)	7·1 (1)
C(22)	0·9422 (6)	0·2541 (3)	1·2148 (5)	8·2 (1)
C(23)	0·8811 (5)	0·2717 (2)	1·0977 (5)	8·3 (1)
C(24)	0·7099 (5)	0·2755 (2)	1·0702 (3)	6·3 (1)
C(25)	0·2899 (4)	0·4958 (2)	1·1154 (3)	4·8 (1)
C(26)	0·1597 (5)	0·4925 (2)	1·1750 (4)	6·9 (1)
C(27)	0·2749 (6)	0·5552 (2)	1·0406 (3)	7·4 (1)
C(28)	0·4384 (5)	0·5032 (2)	1·2005 (3)	5·8 (1)
C(29)	0·1795 (5)	−0·0806 (2)	1·1692 (4)	6·9 (1)

Table 2. *Bond lengths* (Å), *bond angles* (°), *and torsion angles* (°) *involving non-*H *atoms with e.s.d.'s in parentheses*

P(1)—O(2)	1·451 (3)	C(12)—C(17)	1·388 (3)
P(1)—O(3)	1·590 (3)	C(13)—C(14)	1·390 (5)
P(1)—O(4)	1·589 (2)	C(14)—C(15)	1·388 (3)
P(1)—N(5)	1·625 (3)	C(15)—C(16)	1·387 (5)
O(3)—C(6)	1·409 (4)	C(15)—C(25)	1·537 (5)
O(4)—C(12)	1·406 (3)	C(16)—C(17)	1·384 (5)
N(5)—C(18)	1·485 (5)	C(17)—C(18)	1·508 (5)
N(5)—C(19)	1·489 (4)	C(19)—C(20)	1·509 (4)
C(6)—C(7)	1·367 (5)	C(19)—C(24)	1·523 (5)
C(6)—C(11)	1·380 (6)	C(20)—C(21)	1·530 (6)
C(7)—C(8)	1·375 (6)	C(21)—C(22)	1·506 (8)
C(8)—C(9)	1·375 (6)	C(22)—C(23)	1·499 (8)
C(9)—C(10)	1·374 (5)	C(23)—C(24)	1·513 (6)
C(9)—C(29)	1·486 (6)	C(25)—C(26)	1·529 (7)
C(10)—C(11)	1·383 (6)	C(25)—C(27)	1·527 (5)
C(12)—C(13)	1·369 (4)	C(25)—C(28)	1·531 (5)

O(4)—P(1)—N(5)	107·6 (1)	C(12)—C(13)—C(14)	119·5 (2)
O(3)—P(1)—N(5)	105·9 (1)	C(13)—C(14)—C(15)	121·2 (3)
O(2)—P(1)—N(5)	115·7 (2)	C(14)—C(15)—C(25)	123·0 (3)
O(2)—P(1)—O(4)	112·1 (1)	C(14)—C(15)—C(16)	117·4 (3)
O(2)—P(1)—O(3)	115·8 (1)	C(16)—C(15)—C(25)	119·6 (2)
O(3)—P(1)—O(4)	98·1 (1)	C(15)—C(16)—C(17)	122·7 (2)
P(1)—O(3)—C(6)	122·7 (2)	C(12)—C(17)—C(16)	117·9 (2)
P(1)—O(4)—C(12)	122·5 (2)	C(16)—C(17)—C(18)	121·5 (2)
P(1)—N(5)—C(19)	119·2 (2)	C(12)—C(17)—C(18)	120·5 (2)
P(1)—N(5)—C(18)	113·2 (2)	N(5)—C(18)—C(17)	112·3 (2)
C(18)—N(5)—C(19)	119·4 (2)	N(5)—C(19)—C(24)	114·0 (3)
O(3)—C(6)—C(11)	122·1 (3)	N(5)—C(19)—C(20)	110·8 (3)
O(3)—C(6)—C(7)	116·8 (3)	C(20)—C(19)—C(24)	110·6 (3)
C(7)—C(6)—C(11)	121·0 (3)	C(19)—C(20)—C(21)	110·7 (3)
C(6)—C(7)—C(8)	119·0 (4)	C(20)—C(21)—C(22)	110·7 (4)
C(7)—C(8)—C(9)	122·5 (4)	C(21)—C(22)—C(23)	111·8 (4)
C(8)—C(9)—C(29)	122·1 (4)	C(22)—C(23)—C(24)	112·1 (5)
C(8)—C(9)—C(10)	116·8 (4)	C(19)—C(24)—C(23)	109·7 (3)
C(10)—C(9)—C(29)	121·2 (4)	C(15)—C(25)—C(28)	109·2 (3)
C(9)—C(10)—C(11)	122·8 (4)	C(15)—C(25)—C(27)	112·0 (3)
C(6)—C(11)—C(10)	117·9 (3)	C(15)—C(25)—C(26)	108·6 (3)
O(4)—C(12)—C(17)	122·0 (2)	C(27)—C(25)—C(28)	107·3 (3)
O(4)—C(12)—C(13)	116·7 (2)	C(26)—C(25)—C(28)	108·3 (3)
C(13)—C(12)—C(17)	121·2 (2)	C(26)—C(25)—C(27)	111·3 (3)

C(19)—C(20)—C(21)—C(22)	55·7 (5)	N(5)—C(18)—C(17)—C(12)	32·8 (4)
C(20)—C(21)—C(22)—C(23)	−54·2 (6)	C(18)—C(17)—C(12)—O(4)	−0·9 (5)
C(21)—C(22)—C(23)—C(24)	55·3 (6)	C(17)—C(12)—O(4)—P(1)	−7·7 (4)
C(22)—C(23)—C(24)—C(19)	−56·3 (5)	C(12)—O(4)—P(1)—N(5)	−13·7 (3)
C(23)—C(24)—C(19)—C(20)	57·6 (4)	O(2)—P(1)—O(3)—C(6)	66·1 (3)
C(24)—C(19)—C(20)—C(21)	−57·8 (4)	C(18)—N(5)—P(1)—O(2)	170·4 (2)
O(4)—P(1)—N(5)—C(18)	44·2 (2)	O(2)—P(1)—N(5)—C(19)	22·0 (3)
P(1)—N(5)—C(18)—C(17)	−54·4 (3)	O(4)—P(1)—O(3)—C(6)	−174·5 (3)

geometrical calculations, *MOLDRAW* (Ugliengo, Borzani & Viterbo, 1988) for molecular illustrations.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic thermal parameters for non-H atoms are given in Table 1.* Bond lengths, valence angles and selected torsion angles are given in Table 2. A view of the molecule with the atom numbering is shown in Fig. 1. The bond lengths and angles are normal. The bond length P(1)—O(2) of 1·45 (2) Å agrees with those found in other cyclophosphamide, isophosphamide and tropophosphamide compounds (Mikolajczyk, Omelanczuk, Abdukakharov, Miller, Wieczorek & Nojciechowska, 1982). The oxazaphosphorine ring adopts a conformation intermediate between a half-chair and a sofa.

The dihedral angle between the planes C(12)—C(17)—C(18) and O(4)—C(12)—C(18)—N(5) is 26·03° and between O(4)—C(12)—C(18)—N(5) and O(4)—P(1)—N(5) is 161·3°. Puckering parameters for the phosphorinane ring are $q_2 = 0{\cdot}3812$, $q_3 = 0{\cdot}2439$, $Q = 0{\cdot}4526$, $\varphi_2 = 13{\cdot}69$ and $\theta = 57{\cdot}4$. The possibility exists that this ring undergoes conformational changes owing to the steric and electronic demands of its substituents with respect to the substituent on the N (Chandrasekaran

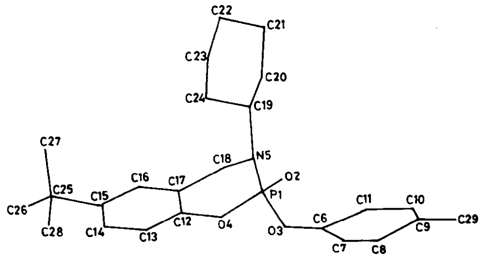

Fig. 1. A view of the molecule with the atom numbering.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles involving H atoms, and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51885 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

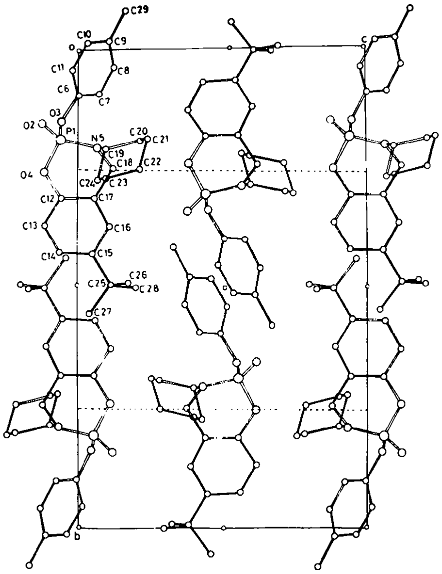

Fig. 2. A perspective view of the molecules in the unit cell.

& Bentrude, 1980) because, firstly, the substituent at N(5) is rotated away by 104·7° and, secondly, the distance O(2)—C(19) is 3·02 (2) Å. The phosphoryl O(2) atom occupies an axial position. The least-squares plane of the 4-methylphenyl ring is inclined at an angle of 126 (1)° to the least-squares plane of the phosphorine ring. The *tert*-butyl and phenyl groups are *trans* to each other and they occupy equatorial positions on the phosphorine ring. The dihedral angle between the two phenyl rings is 47·9°. The cyclohexyl ring adopts a chair conformation with the N(5) atom occupying an equatorial position. Fig. 2 shows a perspective view of the molecules in the unit cell. Molecular packing only involves van der Waals contacts.

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Structure of 9,10-Dihydro-1,9:4,10-diethano-9,10-ethenoanthracene

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Abstract. $C_{20}H_{16}$, $M_r = 256{\cdot}34$, orthorhombic, $Pnm2_1$, $a = 8{\cdot}750\ (1)$, $b = 9{\cdot}419\ (1)$, $c = 8{\cdot}042\ (1)$ Å, $V = 662{\cdot}7\ (1)$ Å^3, $Z = 2$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}30$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}0790$ mm^{-1}, $F(000) = 272$, $T = 293$ K, final $R = 0{\cdot}054$ for 691 observed reflections. The molecule has mirror symmetry in the crystal. All the bond lengths are normal, but the unusual bond angles around the bridgehead suggest that there is much strain in this molecule.

Introduction. The title hydrocarbon, possessing a dibenzobarrelene structure, has recently been synthesized by hydrolysis of *anti*-4-amino-[2.2](1,4)-

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